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**IMPACT SENSITIVITY OF HAN-BASED
LIQUID PROPELLANTS AT
ELEVATED TEMPERATURES****IRVIN C. STOBIE
BRUCE D. BENSINGER
JOHN D. KNAPTON****DTIC
ELECTE
JUN 13 1988**
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<p>A Technoproducts drop weight tester was used to evaluate impact sensitivity of four liquid monopropellants; OTTO II, NOS 365, LP 1845 and LP 1846. The monopropellants were evaluated for impact sensitivity at ambient temperatures and at temperatures in excess of 50°C. The propellants were all relatively insensitive to impact compared to the calibration liquid n-propyl nitrate at ambient temperatures. The impact sensitivity increased slightly for the HAN-based monopropellants at temperatures up to 50 degrees. The impact sensitivity of OTTO II increased dramatically as a function of increased temperature.</p>					
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I. INTRODUCTION

Impact energy tests have been used extensively to evaluate sensitivity of energetic liquids.¹ These tests have been well defined in procedure, and have been performed on a variety of energetic liquids. The drop weight tests are useful as a sensitivity rating device for combustion type environments as well as an indication of behavior under rough handling storage conditions. The HAN-based liquid propellants have been evaluated in other drop weight tests^{2 3} at ambient temperatures, however the tests in the present study were expanded to include tests above the maximum temperature requirements for military ammunition.

II. APPROACH

Impact tests are designed to test compression ignition of propellants in a partially filled chamber. The test procedure is defined in ASTM standard tests D2540-70.⁴ The tests are performed by placing 0.03 ml of the test liquid in a sample cup containing an "O" ring to determine the initial chamber. A 0.41 mm thick stainless steel diaphragm and a piston with a vent orifice are placed on the O ring in the sample cup. The sample chamber is completed by placing the sample cup in a body and assembling with a steel ball and cap as shown in Figure 1. A weight of two kilograms is dropped from a measured height and a positive or negative reaction is recorded. A positive test result is determined by the rupture of the diaphragm and the consumption of the liquid monopropellant. A series of tests are performed on the liquid propellant to determine a height that yields 50% positive results with the 2 kg weight. The ASTM standard states that monopropellants with E50 (defined as the 50% ignition condition) values of 2 kg-cm or less, such as nitroglycerin and diethylene glycol dinitrate, are to be considered sensitive explosives and should be handled with extreme caution.⁴

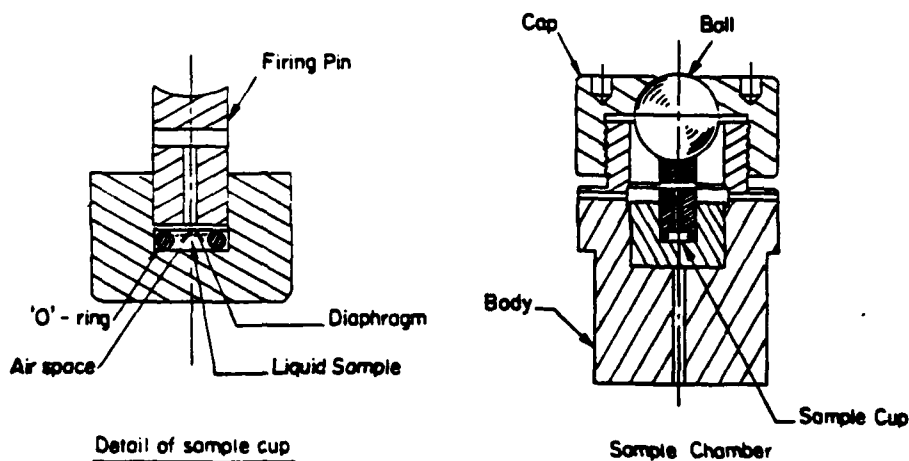


Figure 1. Drop Weight Assembly

III. EXPERIMENTAL

Four monopropellants were chosen for evaluation in this study. OTTO II was evaluated because it has been used extensively in bulk loaded and regenerative liquid propellant guns by many groups. Although Mason et al. report a wide range of the E50 values for OTTO II,⁵ the monopropellant was considered an excellent material for calibration of the drop weight tester and an example of a shock insensitive material. The other three monopropellants were NOS-365, LP 1845, and LP 1846 which were all hydroxyl ammonium nitrate (HAN) based. The NOS-365 monopropellant has been used extensively in bulk loaded firing programs^{6,7} in medium and small caliber guns. The LP 1845 and LP 1846 are considered leading candidates for liquid propellant guns.

In addition to the monopropellant drop weight tests, a series of tests were performed on the liquid n-propyl nitrate, which is used in ASTM D2540 as a reference test liquid. The initial tests of ambient HAN based propellants and the n-propyl nitrate were performed in 1982. An E50 value of 12.8 kg-cm for n-propyl nitrate was measured as compared to the ASTM value of 8.4 kg-cm. This value was considered reasonably consistent with the ASTM value and other reported sensitivities. Subsequent sensitivity tests performed in 1985 and 1986 were also preceded by tests with n-propyl nitrate. No consistent results were noted with three samples of n-propyl nitrate. Subsequent gas chromatography tests indicated the presence of contamination of isopropyl nitrate.⁸

The drop weight tester was standardized by comparing ambient sensitivities of the liquid monopropellants to the earlier 1982 tests where uncontaminated n-propyl nitrate samples were available. The same lots of liquid monopropellants were used throughout the tests. The results at ambient temperatures for all the liquid propellants agreed well with the 1982 ambient tests.

IV. AMBIENT TEMPERATURE TESTS

The liquid propellants were evaluated at 20 degrees plus or minus one degree Centigrade. These results were compared to ambient tests performed by other investigators. Havens reported in Reference 1 that some caution should be used in comparing results from different drop weight tests devices. Cruice (Reference 2) reported sensitivity values above the 100 kg-cm maximum achievable with the instrument we used with a 2 kg drop weight. Attempts to duplicate sensitivity values near 150 kg-cm would have resulted in a positive result, (perforated diaphragm), with only water as the test liquid. The results of the ambient tests along with results from other sources are shown in Table 1. The monopropellant LP 1846 had not been previously evaluated in a drop weight tester. The 50% ignition values of 98 kg-cm for OTTO II and NOS-365 indicate highly shock insensitive materials. Only 33% positive tests were noted for LP 1845 at the maximum drop weight, and LP 1846 sensitivity was beyond the capabilities of the instrument.

TABLE 1. Ambient Temperature Monopropellant Drop Weight Results

PROPELLANT	EARLIER TESTS		PRESENT STUDY
	50% IGNITION kg-cm	REFERENCE	% IGNITION %, kg-cm
OTTO II	8.5-70	5	50%, 98
NOS-365	>100	3	50%, 98
LP 1845	152	2	33%, 100
LP 1846	---		>100

V. ELEVATED TEMPERATURE TESTS

The elevated temperature tests were performed by keeping the sample cup, piston and liquid propellant in a temperature conditioning oven prior to firing. The temperature was recorded immediately before the weight was dropped by means of a thermocouple on the sample cup. Table 2 summarizes the sensitivity results and the temperature ranges for the four monopropellants.

TABLE 2. Ambient Results and Temperature Ranges and Results for Elevated Temperature Drop Weight Tests.

PROPELLANT	TEMPERATURE	50% IGNITION
	°C	kg-cm
OTTO II	ambient	98
	34-54	<52
NOS-365	ambient	98
	34-49	93
LP 1845	ambient	(33%-100)
	37-57	88
LP 1846	ambient	>100
	31-53	96

The results with OTTO II at elevated temperatures were somewhat confusing. The initial temperature conditioned tests yielded results that were borderline positive reactions such as liquid residue in the cup and minimum noise from the reaction. As more temperature conditioned tests were performed, the reactions became more complete even at shorter drop heights. It was speculated that the temperature conditioning had an effect on the propellant. A new sample of OTTO II was therefore tested. The ambient sensitivity was checked with previous

tests and the elevated temperature tests were performed. For this sample of propellant the conditioning temperatures were monitored to assure that the sample was never above 70°C. Once again the propellant apparently became more sensitive as a function of time in the conditioning over. The results of the monitored temperature conditioned tests are shown in Table 3.

TABLE 3. Sensitivity Results of OTTO II Conditioned at Temperatures Less Than 70°C

TESTS	ENERGY kg-cm	TEMPERATURE °C	POSITIVE RESULTS
1-5	96-100	38-40	3
6-10	80-92	38-45	4
11-15	68-74	35-45	4

LP 1846 was chosen for further evaluation at the monitored temperature conditions. This propellant, along with LP 1845, is considered a likely candidate for future weapons testing. LP 1845 differs from LP 1846 by three percent less water. It is interesting to note that the sensitivity for both the ambient and elevated tests indicated slightly more sensitivity for LP 1845 as compared to LP 1846.

A sample of LP 1846, along with the sample cup and piston, was conditioned for at least 30 minutes to temperatures up to 58°C. Fourteen tests were performed at drop weight heights of 100 kg-cm at temperatures between 32° and 42°C. Three of the fourteen tests were recorded as positive results. The conditioning temperature was raised to a value less than 78°C for the next series of tests. There were five positive tests noted at 100 kg-cm values and no positive reactions in four attempts at 98 kg-cm. A new sample of LP 1846 was conditioned for at least 30 minutes at temperatures up to 82°C. The results of this conditioning are shown in Table 4. The obvious change in sensitivity in the 18 June tests are noted. The sample was sealed and stored and the 20 June ambient tests were performed with no apparent change in sensitivity. The sample was then conditioned to a temperature that had little effect on the previous sample. The 20 June tests 6-15 revealed the unusual sensitivity shown in Table 4. A Carl Fisher titration of the conditioned sample and a sample from the same lot of propellant was performed. The conditioned sample contained 15.05% water, while the unconditioned sample from the same lot contained 19.32% water.⁹ The dramatic shift in sensitivity between the 78° and 82°C was apparently caused by a change in the chemical composition of the propellant.

TABLE 4. LP 1846 Drop Weight Sensitivity After Being Conditioned at Temperatures Less Than 82°C.

TEST NOS.	TEMPERATURE °C	ENERGY kg-cm	RESULTS
18 June 51-57	54-62	100	2/4 Positive
		98	1/1 Positive
		96	1/1 Positive
		80	1/1 Positive
20 June 1-5	22	100	0/5 Positive
20 June 6-15	44-48	100	1/1 Positive
		94	2/2 Positive
		92	2/4 Positive
		90	0/2 Positive

VI. CONCLUSIONS

1. The four liquid monopropellants are insensitive to the maximum drop weight condition at ambient temperatures.
2. The drop weight sensitivity increased dramatically for the elevated temperature tests with OTTO II. Even when conditioning temperatures were held under 58°C, the sensitivity remained quite high.
3. The drop weight sensitivity increased slightly for the three HAN based monopropellants at temperatures up to 57°C.
4. The sensitivity of LP 1846 increased dramatically at conditioning temperatures between 78 and 82°C. This shift in drop weight sensitivity is believed to be due to the loss of water in the liquid propellant.

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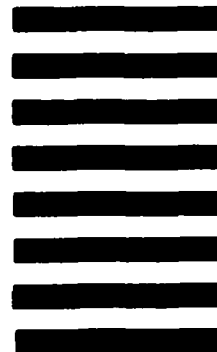


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